Miniaturized Scanning Electron Microscope for In-Situ Planetary Studies

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ABSTRACT

The exploration of remote planetary surfaces calls for the advancement of low-power, low-mass, highly-miniaturized instrumentation. Multi-functional instruments of this nature will prove to be particularly useful in preparation for human return to the moon, and in exploring increasingly remote locations in the Solar System. To this end, our group has been developing a miniaturized Scanning Electron Microscope (mSEM) capable of remote investigations of mineralogical samples through in-situ topographical and chemical analysis on a fine scale. Specifically, the fabrication and testing of a proof-of-concept assembly has begun, and consists of a cold-field-emission electron gun and custom high-voltage power supply, electrostatic electron-beam focusing column, and scanning-imaging electronics plus backscatter electron detector.

The functioning of an SEM is well known: an electron beam is focused down to nanometer-scale onto a given sample resulting in emissions such as backscattered and secondary electrons, x rays, and visible light. Raster-scanning the primary electron beam across the sample results in a fine-scale image of the surface topography (texture), crystalline structure and orientation, with accompanying elemental composition.

The flexibility in the types of measurements the mSEM is capable of makes it ideally suited for a variety of applications. The mSEM is appropriate for use on multiple planetary surfaces, and for a variety of mission goals (from science to non-destructive analysis to in-situ resource utilization). The current status of the development and potential mSEM applications for planetary exploration are summarized here.

This effort is funded through the NASA Research Opportunities in Space and Earth Sciences - Planetary Instrument Definition and Development Program (PIDDP).
INTRODUCTION

Mankind has always been fascinated with the heavenly bodies that comprise our Solar System. We have made it a point to explore and understand these bodies in an attempt to satisfy a collective curiosity regarding the origin and evolution of the Solar System. Whether it is the moon, Mars, or other planetary bodies that we wish to explore, suitable instrumentation will be needed to deliver the information sought.

With a focus on functionality, low-power, and miniaturization our group has initiated development of a miniaturized Scanning Electron Microscope (mSEM) capable of nanometer-scale resolution, in-situ topographical imaging, and compositional Energy Dispersive x-ray Spectroscopy (EDS) of uncoated natural and synthetic samples. The diversity of measurements that can be made with an SEM (and particularly one with environmental mode capabilities) makes it an ideal instrument for a variety of planetary investigations. This is highlighted by the fact that there has been multiple previous mini-SEM developments intended for various missions.

The concept of a miniaturized SEM is not a new one. The SEM and Particle Analyzer (SEMPA), developed in the late 1980’s specifically for the Comet Rendezvous Asteroid Flyby satellite (since canceled), achieved ~40nm resolution, operated on a relatively low power of 22W, and weighed roughly 12kg (Conley et al., 1983; Albee & Bradley, 1987). Utilizing current technologies, the design presented here will permit an even smaller, lighter, lower-power version that is easily adaptable to a variety of missions. Additional efforts in this field have produced a range of results concerning the development of a mini-SEM or miniature electron focusing column (Khursheed, 1998; Gross, 2005; Callas, 1999; Roberts et al., 1997; Yabushita et al., 2007).

For the sake of simplicity, our development program is limited to a lunar version of the mSEM. While many of the components are directly translatable to other environments - such as Mars - other components will need some modification (i.e. a vacuum system). The lunar mSEM instrument goals are summarized in Table 1.

Table 1. Below is a list of the desired mSEM characteristics. The overall system dimensions include all support electronics. The power estimate is based on the current high-voltage power-supply design.

<table>
<thead>
<tr>
<th>Parameters</th>
<th>Goals</th>
</tr>
</thead>
<tbody>
<tr>
<td><strong>Electron Gun/Column/Scanning Assembly</strong></td>
<td></td>
</tr>
<tr>
<td><strong>Size</strong></td>
<td>~5 cm long, &lt;2 cm diam.</td>
</tr>
<tr>
<td><strong>Mass</strong></td>
<td>&lt; 50g</td>
</tr>
<tr>
<td>Imaging Resolution</td>
<td>&lt; 100 nm</td>
</tr>
<tr>
<td>Maximum Accelerating Voltage</td>
<td>10 kV</td>
</tr>
<tr>
<td>Maximum Field of View</td>
<td>5-10 mm square</td>
</tr>
<tr>
<td>Sample Preparation</td>
<td>Minimal – No Coatings</td>
</tr>
<tr>
<td>Overall System Dimensions</td>
<td>&lt; 20 cm x 10 cm x 10 cm</td>
</tr>
<tr>
<td>Total Power</td>
<td>15 W</td>
</tr>
</tbody>
</table>
Efforts to date have produced proof-of-concept mSEM components (electrostatic electron focusing column and scanning system) that have resulted in a first focused image (Fig. 1). Progress has been made on the remaining proof-of-concept components which include a custom electron gun and associated power-supply system, and complete scanning/imaging system plus backscattered electron (BSE) detector. The final instrument will also include an EDS (custom silicon drift detector), which is outside of the scope of the current effort.

This work is a collaborative effort between NASA MSFC, Advanced Research Systems, the University of Alabama Huntsville, the University of Tennessee Knoxville and Case Western Reserve University, with contributions also from Johns Hopkins Applied Physics Laboratory.

![Image of mSEM components](image)

**Figure 1:** The image on the left shows three proof-of-concept mSEM components that have been fabricated and tested. The resulting imaging resolution (of a few microns) is much larger than that of the final version mSEM, due to the relatively large emission region that results from using a thermionic electron gun compared to that of a FEG (for which the focusing column was designed).

**LUNAR SURFACE SCIENCE**

The return of humans to the Moon is a major step in NASA’s plan for exploration. It is imperative that preparations be made for the science and engineering challenges that will be an intrinsic part of this vision, especially as it pertains to the study of lunar regolith.

Although considerable research has already been performed on lunar regolith collected during the Apollo and Luna Missions, this sampling represents less than ten percent of the lunar surface (Heiken et al., 1991; Jolliff et al., 2006). The successful realization of a lunar outpost will require significant expansion of our knowledge relating to lunar regolith (pertinent to in-situ resource utilization (ISRU); Chambers et al., 1994; Chambers et al., 1995). The mSEM’s ability to permit in-situ morphological and chemical characterization of lunar regolith will minimize the need for sample return and allow for the differentiation of unique samples tagged for Earth return.

Morphological and chemical characterization of lunar regolith in laboratories on Earth has been routinely accomplished using SEMs and EDS (McKay et al., 1991).
The utility of the mSEM will be discussed relative to its ability to determine mineral and glass compositions, particle size distribution and morphologies, types of rocklets (~1-10mm rock chips) and their mineralogies, and general characteristics of lunar regolith components. These data relate to the science of soil formation and its utilization for resources (e.g., production of oxygen: Taylor & Carrier, 1992; Taylor & Carrier, 1993) from both the Moon's Maria and Highland terrains.

Further reading on the benefits of a lunar mini-SEM can be found in our sister paper by Thaisen et al. (2010) of these Proceedings.

STUDIES OF EXPOSED MATERIALS

In addition to science studies, the mSEM would be a powerful tool for studying the deleterious effects of the lunar environment. High-resolution images can capture surface corrosion, micrometeorite-impact effects, UV-induced material degradation, and the effects of stresses imposed by the extremes of temperatures present on the lunar surface. These effects can show up as a loss in thermal, electrical and optical properties and diminished structural integrity. The mSEM would allow the analysis of fracture surfaces to identify common failure modes, such as overload, fatigue, and creep (McEvily, 2001). Coincident chemical analysis would allow for identification of residues and reaction products formed during lunar operations. For long-term human habitation, such studies are vital to ensure selection of appropriate materials.

It is worth noting that these studies are especially important in the harsh Martian surface environment. The mSEM would be ideal for carrying out contamination and corrosion studies; and similar studies of how manufactured materials react or degrade on the surface of Mars – essential for a human presence there. The Martian environment is 1) highly corrosive, due to the presence of a strong oxidant capable of rapid destruction of organic compounds; 2) highly abrasive due to suspended, micron-scale dust driven at high velocities by seasonal dust-storms; 3) highly charged due to static voltages generated by the movement of this dust in an effectively nonconductive atmosphere; and 4) photochemically active because of the unfiltered high UV flux. The mSEM provides a means to directly and empirically study corrosive processes, in situ, and their associated risks through the examination of purposefully exposed materials.

METHODOLOGY

A standard SEM takes up the space of a large desk, weighs about half a ton, and requires kilowatts of power to operate. The transformation of an SEM to a planetary exploration instrument necessitates considerable miniaturization. Our approach has involved a complete rethinking of the basic SEM design that has prevailed for over 60 years, and makes use of a combination of innovative materials and construction techniques, which have been validated in our prototype. The nature of the lunar
environment (i.e., high ambient vacuum of \( \sim 10^{-12} \) torr) allows for simplification of the overall instrument design that can then be modified for use on Mars.

The main components of an SEM include: an electron gun; electron focusing lenses; a deflection/scanning system; sample chamber or interface, electron and x-ray detectors, and a vacuum system. Electrons generated in the gun propagate through the electron-optics assembly (consisting of precisely placed apertures and an electrostatic lens) and are focused onto the sample. The scanning coils rapidly raster this focused beam across the sample to create the subsequent image and characteristic x rays. The development of proof-of-concept components has begun, and includes: a custom electron cold-Field-Emission Gun (FEG), a focusing column (with electrostatic lens system), and scanning/magnification system. Using a novel electron-focusing column and scanning system combined with an off-the-shelf thermionic electron gun and support electronics from a commercial SEM, a first-focused image of a copper-grid standard has been obtained (Fig. 1).

Because a thermionic cathode was used for the initial electron-focusing column testing, the resulting imaging resolution did not meet the goal of <100 nm. Since that time, a cold FEG, for which our column was designed for use with, has been successfully fabricated and tested.

FIELD EMISSION ELECTRON GUN

The electron gun being developed is a cold-field emitter that utilizes an off-the-shelf Hitachi tungsten cathode. A Butler-like triode configuration is employed, and consists of a field-emitter tip followed by a first and second anode (Butler, 1966). A large applied field between the field-emitter tip and the first anode causes electrons to tunnel out of the tip (Gomer, 1961). These electrons are then accelerated towards the second anode, which is typically at ground. The accelerating voltage of the gun is defined as the voltage between the field emitter tip (i.e., cathode tip) and the second anode. The extraction voltage is the potential difference between the cathode tip and first anode. One can use the Fowler-Nordheim equation (Fowler & Nordheim, 1928), in modified form (Murphey & Good, 1956) and restated in more general terms by Forbes 1999, to describe the relationship between emission-current density, \( J \), and local field at the surface, \( F \):

\[
J = \lambda a \phi^{-1} F^2 \exp (- \mu b \phi^{3/2} F^{-1}),
\]

\[
a = \frac{e^3}{8 \pi \hbar} = 1.541 \times 10^{-6} \text{ } eV V^2,
\]

\[
b = \frac{4}{3} \left( \frac{2m_e}{e \nabla} \right)^{1/2} = 6.830 \times 10^9 \text{ } eV^{3/2} V m^{-1},
\]

where \( \lambda \) and \( \mu \) are correction factors, \( a \) and \( b \) are universal constants, and \( \phi \) is the work function of the emitter, \( e \) is elementary charge, \( h \) is Planck’s constant, \( \nabla = h/2\pi \), and \( m_e \) is the mass of an electron. A small increase in the extraction voltage (related to \( F \)) will result in a large increase in the total emission current. If the potential difference between the cathode and first anode becomes too large, the fine cathode tip...
can be damaged. Precise control of the extraction voltage is required to avoid this and to maintain emission current stability.

MECHANICAL CONFIGURATION

The first generation of the FEG design suffered unexpected breakdown in the cathode chamber that led to breakdown in the high-voltage supplies. The second generation features a more robust design that will be refined (further miniaturized) once the control system has been proven.

Key features of this electron gun include:

• Fine horizontal alignment mechanism for centering the cathode tip to first anode.
• Vertical-alignment mechanism, accomplished via a finely threaded, graduated fitting which allows the field-emitter tip to be placed at the desired distance from the first anode, to within ~10\(\mu\text{m}\).
• Cathode tip to first anode alignment verification by an optical microscope, before the second anode is attached.
• Ceramic spacers to electrically isolate the tungsten cathode from the first anode and to isolate the second anode from the first.

The first anode is an off-the-shelf platinum Hitachi aperture and the second anode is a stainless-steel cap with a small hole drilled into it. A Faraday cup is attached directly behind the second anode to measure the current. Figure 2 illustrates this design.

Figure 2. Diagram depicting the main components for our electron gun. The image on the bottom-right corner is the assembled gun, with Faraday cup attached just after the second anode.
EMISSION CURRENT CONTROL

A high-voltage power supply (HVPS) assembly and control system for the electron gun have been developed by collaborators at the University of Alabama Huntsville and with input from additional team members. The electron gun operates at a maximum accelerating voltage of 10kV.

Main Components/Features include:
• Three compact off-the-shelf switching power converters procured from Ultravolt, Inc. Two of these generate the voltages on the cathode and first anode, and the third functions as an isolator, to allow the first anode power supply to be ground referenced to the cathode high voltage.
• Power for the high-voltage supplies, provided by a commercial laboratory supply for testing. This supply can be easily replaced by a battery or alternate portable source at a later time (total power to run these three supplies is ~10 W).
• Compact design of HVPS system. The entire HVPS system (without any repackaging of the Ultravolt units or electronics boards) fits easily inside a small enclosure (~8” x 4” x 4”). Further miniaturization is possible with custom-designed power supplies and/or repackaging of these supplies and electronics.
• Emission-current control and monitoring; currently accomplished with the use of potentiometers and multimeters. Eventually, microcontrollers will be used. The user dials-in the desired emission current and accelerating voltage, and the potential on the first anode is automatically adjusted to maintain the desired current. Maximum accelerating voltage is 10kV.
• A flashing circuit, developed to maintain the cathode tip atomically clean.

TESTING & DISCUSSION

The electron gun has, thus far, only been tested with a blunted Hitachi tungsten FE cathode, vertically aligned to within 50µm of the first anode. The entire gun assembly sits inside of a large spherical vacuum chamber (capable of operating at high 10^-10 torr). Figure 3 shows SEM images of a sharpened tip versus a blunted one. The blunted tip – created by accidentally ripping the tip off, by placing it in a high electrical field – has a much larger tip radius than the sharpened one. The effect of this is a diminished total-emission current. These field emitters are relatively expensive and easily damaged – especially in the presence of high-voltage breakdown in the gun or even in the high-voltage supplies themselves. This preliminary testing has allowed validation of the safety of all instrument components and of the high-voltage assembly itself (as parts of it are floating at 10kV).
Figure 3. Images taken using a commercial FEI Quanta 600 FEG SEM and depicting the FE cathodes at various magnifications. Image A) shows a complete tungsten cathode structure; B) is a magnification of the circled region in Image A; C) is a magnification of the circled region in Image B., and clearly shows the sharpened tip structure. Image D) is of a blunted tip structure (rotated 180°). Images are courtesy of G. Jerman (NASA MSFC).

With this blunted tip installed in the electron gun, it was possible to successfully and repeatedly regulate emission current from the cathode. Table 2 shows results from three input currents, I_in. The current on the second anode surface, I_a2, was recorded along with the current seen by the Faraday cup, I_F and the extraction voltage, V_e (i.e. the voltage between the cathode and first anode). The accelerating voltage was 10.13kV and the test chamber pressure was ~1x10^-8 torr.

Table 2. Three trials showing the input current to the cathode (I_in), extraction voltage applied to maintain this set current (V_e), and resulting currents monitored on the second anode (I_a2) and in the Faraday cup (I_F).

<table>
<thead>
<tr>
<th>Parameter</th>
<th>Trial 1</th>
<th>Trial 2</th>
<th>Trial 3</th>
</tr>
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<tbody>
<tr>
<td>I_in (µA)</td>
<td>0.52</td>
<td>1.0</td>
<td>2.4</td>
</tr>
<tr>
<td>I_a2 (µA)</td>
<td>0.22</td>
<td>0.46</td>
<td>1.12 - 1.2</td>
</tr>
<tr>
<td>I_F (nA)</td>
<td>0.4</td>
<td>0.8</td>
<td>2</td>
</tr>
<tr>
<td>V_e (kV)</td>
<td>3.44</td>
<td>3.59</td>
<td>3.78 - 3.88</td>
</tr>
</tbody>
</table>
The ranges in Trial 3 are due to the fact that emission was not stable. This is likely due to the fact that a blunted tip was used rather than a sharp one. The data that are reported here are approximate values, as these trials were intended for system checkout before a sharpened tip was installed in the electron gun. Further, these results are somewhat polluted due to leakage current between the regulation circuit and the power supplies.

From Table 2 it is apparent that as the user input current is increased, the extraction voltage automatically increases accordingly. It is also evident, yet not surprising, that the majority of the current produced in the cathode goes onto the first and second anode surfaces, rather than into the Faraday cup. To successfully image and perform EDS, typically a beam current of only a few to 10 nA is required (e.g. Erdman et al., 2009). With a sharpened tip installed, much more of the current should make it through the second anode.

In parallel with these tests, simulations are being conducted on the electron gun configuration to determine the presence and effects of aberrations. These models will be used for optimization of the next iteration. The program being used for this is Charged Particle Optics software from Scientific Instrument Services, Inc.

**INSTRUMENT CONTROL & IMAGING**

In order to provide a configurable base control system for the mESEM a custom designed embedded microcontroller system is currently under construction. To provide flexibility this system is designed to provide communications, command and control over a single USB 2.0 link to a host computer. The host computer can thus be a commercial desktop or laptop computer running Windows, Mac OS or a Unix variant for testing and direct human use. The host computer could also be a high capability embedded computer providing autonomous instrument control and handling telemetry of instrument control and data return for robotic missions.

The mSEM controller utilizes the USB 2.0 provisions for multiple ‘endpoints’, or communication channels, to receive control information from the host computer and to send status information to the host computer. Simultaneously, a high speed FIFO and serial control structure allows for 480Mbit transfer rates of image and detector data to the host computer. Electron beam scanning and image transmission is tightly integrated with the controller using a self contained state machine, which is part of the embedded microcontroller, along with custom circuitry to generate the required waveforms, digitize the detector data and send the image data to the host computer with little intervention from the embedded microcontroller.

The embedded microcontroller is then free to monitor and control all of the functions it oversees as well as communicate this data to the host computer. The embedded controller has two high speed RS-232 channels and an eight bit bi-directional bus with fully decoded device addressing available for control and monitoring of other functions such as the detectors, electron gun, focusing, sample handling and vacuum
control. The embedded microcontroller will also provide the first level of exception handling and system diagnostics.

The host computer is responsible for high level control of the mSEM through simple commands to the embedded microcontroller. It will also reconstruct the resultant detector data into images suitable for viewing and analysis. For direct human use an easy to use GUI will provide for complete operation of the mSEM. For remote applications, various levels of autonomous control and analysis can be programmed.

**DETECTORS**

At present, two detector systems are being contemplated for the mSEM – a four-quadrant BSE detector and a SDD EDS x-ray micro-analyzer system. As mentioned before the SDD system is beyond the purview of the current project but initial testing will be probably be done with a commercially available system and later expanded to a complete custom design appropriate to the size, weight and power requirements of the mSEM.

Negotiations are currently underway with the manufacturer of a novel BSE detector. As this detector will be small enough to fit well with the mSEM only the physical mounting and electronic amplification of the detector signal need be addressed.

Construction and testing of the BSE detector circuits will begin with the completion of the embedded controller and continue through prototyping of the full system.

**SUMMARY & FUTURE WORK**

Progress of the mSEM development includes successfully fabricating and testing an electron-focusing column (electrostatic lens) and obtaining our first focused image. An electron-gun assembly has been fabricated and tested with a blunted Hitachi cathode and it was possible to successfully regulate the desired beam current using a newly-developed HVPS system. For the next round of testing a sharpened cathode tip will be inserted into the gun assembly to fully characterize the gun operation in terms of total beam current. Following the successful completion of this testing, the column/scanning system will be mated with the electron gun assembly along with the imaging/BSE detector assembly for final testing and characterization.
REFERENCES


